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# *Indian Standard*
# METHODS FOR SAMPLING OF CRYOLITE AND ALUMINIUM TRIFLUORIDE

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**INDIAN STANDARDS INSTITUTION**
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 1

*Price Rs* 4·00 Gr 3

*September* 1968

# *Indian Standard*

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( *Continued on page* 11 )

# *Indian Standard*

# METHODS FOR SAMPLING OF CRYOLITE AND ALUMINIUM TRIFLUORIDE

## 0. FOREWORD

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 12 August 1968, after the draft finalized by the Methods of Sampling Sectional Committee had been approved by the Structural and Metals Division Council.

**0.2** The demand for cryolite and aluminium trifluoride in the continuously expanding aluminium industry have been steadily growing in India during the last decade. The natural cryolite, which is mostly imported, has a wide range of other uses, such as whitener for enamels in the ceramic industry, as an opacifier in glasses, for bonding of grinding wheels and abrasives and also as an ingredient in welding rod coatings. The manufacture of synthetic cryolite has also started in the country. At this stage of development and expansion for the exploitation of these ores, it was, therefore, felt desirable that due consideration be given to the sampling procedures which will help in proper and objective evaluation of the quality of these minerals.

**0.3** Taking into consideration the views of the producers, testing authorities and consumers, the Sectional Committee responsible for the preparation of this standard felt that it should be related to the sampling procedures generally followed in the country in this field.

**0.4** This standard is one of a series of Indian Standards on sampling of ores and aggregates. Other standards on methods of sampling in this series are:

| | |
|---|---|
| IS : 1405 - 1966 | Iron ore (*first revision*) |
| IS : 1449 - 1961 | Manganese ore |
| IS : 1811 - 1961 | Foundry sands |
| IS : 1999 - 1962 | Bauxite |
| IS : 2109 - 1962 | Dolomite, limestone and other allied materials |
| IS : 2245 - 1962 | Quartzite |
| IS : 2246 - 1963 | Fluorspar (fluorite) |
| IS : 4156 - 1967 | Barytes |
| IS : 4166 - 1967 | Ilmenite and rutile. |

**0.5** This standard contains clauses **7.2.1** and **8.1** which call for an agreement between the purchaser and the supplier.

**0.6** In reporting the results of a test or analysis, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

## 1. SCOPE

**1.1** This standard lays down the procedure to be followed in collecting and preparing samples from a lot in order to determine the moisture content and the chemical composition of cryolite/aluminium trifluoride in the lot. It details the procedure for sampling of the mineral from conveyors, wagons, ship's holds and stock piles.

**1.1.1** This standard also includes a method for reporting the quality of the bulk of the mineral sampled.

## 2. TERMINOLOGY

**2.0** For the purpose of this standard the following definitions shall apply.

**2.1 Lot/Stock Pile** — The quantity of cryolite/aluminium trifluoride offered for inspection at one time; a lot may consist of the whole or a part of the quantity ordered for.

**2.2 Sub-lot** — The quantity of mineral in each of the groups into which a lot/stock pile is divided for the purpose of sampling.

**2.3 Increment** — The quantity of mineral obtained by a sampling device at one time from a lot or a sub-lot.

**2.4 Gross Sample** — The quantity of mineral obtained by mixing together all the increments collected from the same sub-lot.

**2.5 Laboratory Sample** — The quantity of mineral obtained by reducing a gross sample following a specified procedure and intended for laboratory testing.

**2.6 Moisture Sample** — A laboratory sample meant exclusively for the determination of moisture content.

## 3. SAMPLING FROM CONVEYORS

**3.1 Sub-lot** — For the purpose of sampling, the quantity of mineral to be discharged over a conveyor shall be considered as divided into the number of sub-lots specified in Table 1.

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*Rules for rounding off numerical values ( *revised* ).

### TABLE 1 NUMBER OF SUB-LOTS INTO WHICH A STOCK PILE/LOT IS TO BE DIVIDED

( *Clauses* 3.1, 4.1, 5.1, 6.1.1 *and* 6.2.1 )

| Weight of the Lot ( Tonnes ) | No. of Sub-lots |
|---|---|
| Up to 100 | 1 |
| 101 ,, 250 | 2 |
| 251 ,, 500 | 3 |
| 501 ,, 1 200 | 4 |
| 1 201 ,, 2 500 | 5 |
| 2 501 and above | 6 |

**3.1.1** While the quantity of cryolite/aluminium trifluoride constituting a sub-lot is moving on the conveyor, the increments shall preferably be taken across the full width of the stream at the point of discharge with the help of a suitable sampling implement. The number of increments in such a case shall be approximately one-sixth of the number of increments as specified in Table 2 since these increments are usually of larger weights.

If it is not possible to sample satisfactorily at the point of discharge, the number of increments specified in Table 2 may be drawn from the moving belt itself. In this case, the increments shall be collected from the centre and the left and right sides of the belt along the same width. To ensure that very small material is also correctly obtained in the sample, the scoop should sweep the bottom of the conveyor.

### TABLE 2 NUMBER OF INCREMENTS TO BE COLLECTED FROM A SUB-LOT

( *Clauses* 3.1.1, 4.1.1, 5.1.1, 6.1.1.1 *and* 6.2.1.1 )

| Weight of Sub-lot ( Tonnes ) | Number of Increments | |
|---|---|---|
| | Cryolite* | Aluminium Trifluoride† |
| Up to 100 | 40 | 30 |
| 101 ,, 200 | 56 | 42 |
| 201 ,, 300 | 68 | 52 |
| 301 ,, 400 | 80 | 60 |
| 401 ,, 500 | 88 | 66 |
| 501 ,, 600 | 96 | 72 |
| 601 and above | 104 | 78 |

*For cryolite { Weight of each increment = 0·5 kg. Sampling scoop of capacity 0·5 kg shall be used.

†For aluminium trifluoride { Weight of each increment = 0·2 kg. Sampling probe of capacity 0·2 kg shall be used.

**3.2 Gross Sample** — All the increments collected from the same sub-lot shall be mixed together to constitute a gross sample.

**3.3** Thus, a lot will be represented by as many gross samples as the number of sub-lots into which it is divided. Each gross sample shall be processed further individually in accordance with **7** and tested in accordance with **8**.

## 4. SAMPLING FROM WAGONS

**4.0** Representative samples of cryolite/aluminium trifluoride may be collected either during loading or unloading of wagons.

**4.1 Sub-lots** — All the wagons in the lot shall be divided into the number of sub-lots of approximately equal weight specified in Table 1.

**4.1.1** *Sampling During Loading and Unloading* — For sampling cryolite/aluminium trifluoride when it is being loaded into or unloaded from wagons, the number of increments specified in Table 2 shall be collected at regular intervals during the whole period of loading or unloading each sub-lot. The increment of 0·5 kg for cryolite or 0·2 kg for aluminium trifluoride shall be collected from one or more grabs, baskets, bags or packages.

**4.2 Gross Sample** — All the increments collected from the same sub-lot shall be mixed together to constitute a gross sample.

**4.3** Thus, a lot will be represented by as many gross samples as the number of sub-lots into which it has been divided. Each gross sample shall be processed further individually in accordance with **7** and tested in accordance with **8**.

## 5. SAMPLING FROM SHIPS

**5.1 Sub-lots** — The quantity of mineral in the ship's hold, constituting a lot, shall be divided into the number of sub-lots of approximately equal weight specified in Table 1.

**5.1.1** The number of increments to be collected from a sub-lot shall depend upon the quantity of cryolite/aluminium trifluoride in the sub-lot in accordance with Table 2. The increments shall be collected at regular intervals during the whole period of loading or unloading the material in the sub-lot. The increment, each of weight 0·5 kg for cryolite or 0·2 kg for aluminium trifluoride, shall be collected from one or more grabs, baskets used for loading or unloading.

**5.2 Gross Sample** — All the increments from the same sub-lot shall be mixed together to constitute a gross sample.

**5.3** Thus, a lot will be represented by as many gross samples as the number of sub-lots into which it has been divided. Each gross sample shall be processed further individually in accordance with **7** and tested in accordance with **8**.

## 6. SAMPLING FROM STOCK PILES

**6.0** Sampling of stock piles shall be done by any one of the methods described in **6.1** and **6.2** preferably by the former method.

### 6.1 Sampling During Loading or Unloading of Stock Piles

**6.1.1** *Sub-lots* — The quantity of cryolite/aluminium trifluoride to be loaded into or unloaded from a stock pile shall be considered as divided into the number of sub-lots of approximately equal weight specified in Table 1.

**6.1.1.1** While loading or unloading the quantity of mineral constituting a sub-lot, the number of increments specified in Table 2 ( depending upon the quantity of mineral in the sub-lot ) shall be collected at regular intervals during the whole period of loading or unloading the sub-lots. The increment, each of weight 0·5 kg in the case of cryolite or 0·2 kg for aluminium trifluoride, shall be collected from grabs, baskets, bags or packages.

**6.1.2** *Gross Sample* — All the increments collected from the same sub-lot shall be mixed together to constitute a gross sample.

### 6.2 Sampling a completed Stock Pile

**6.2.1** *Sub-lots* — Mineral in the stock pile shall be divided into the number of sub-lots of approximately equal weight specified in Table 1 by suitably marking the line of demarcation on the surface of the stock pile. The surface of stock pile shall be levelled and the height of the stock pile shall never exceed 1·0 metre.

**6.2.1.1** The number of increments to be collected from a sub-lot shall depend upon the quantity of the mineral in the sub-lot in accordance with Table 2. For collecting these increments each of weight 0·5 kg for cryolite or 0·2 kg for aluminium trifluoride an equal number of points shall be located on the longitudinal parallel lines 1·0 m apart over the whole surface. The point to be so located shall be evenly distributed over all the lines. At each of the points a hole of 40 cm depth shall be made from where increments shall be taken with the scoop. When the stock pile is made up of bags or packages filled with the mineral, at each point as selected above, one bag or package shall be collected. From each bag or package one increment of weight 0·5 kg for cryolite or 0·2 kg for aluminium trifluoride shall be taken.

**6.2.2** *Gross Sample* — All the increments collected from the same sub-lot shall be mixed together to constitute a gross sample.

**6.3** Thus, a lot will be represented by as many gross samples as the number of sub-lots into which it has been divided. Each gross sample shall be processed further individually in accordance with **7** and tested in accordance with **8**.

## 7. REDUCTION OF A GROSS SAMPLE

**7.1** The gross samples from sub-lots of naturally bonded minerals are likely to contain bigger size than the actual grain sizes. So the gross sample shall be crushed below 5·0 mm. The crushed mineral is well mixed and reduced in stages to 6 kg and the moisture sample for determination of moisture content and the laboratory sample for determination of chemical composition are obtained according to the procedure shown in Fig. 1. For reduction at various stages the method described in **7.1.1** or **7.1.2** shall be followed.

**7.1.1** *Reduction by Coning and Quartering* — The crushed material shall be well mixed and dropped so as to form a cone-shaped pile. Care shall be taken to drop each scoopful exactly over the same spot so that the central axis of the cone is not slackened and a regular distribution of grains and fines will result. After the cone is formed, it shall be flattened by pressing the apex with the smooth surface of a flat shovel. Then it is cut into quarters by two planes which intersect at right angles along the axis of the cone. The bulk of the sample is reduced by rejecting any two diagonally opposite quarters.

**7.1.2** *Reduction by Using Sample Splitter or Riffle Sampler* — Mechanical quartering is best done with a riffle. After each crushing, the mineral shall be well mixed and poured into the riffle. This process shall be repeated using different sizes of riffles according to the size of the mineral.

**7.2** The material in each moisture sample and laboratory sample shall be thoroughly mixed and divided into three approximately equal parts. A set consisting of material from each of the moisture sample and laboratory sample drawn from a sub-lot shall be given to the purchaser, another such set shall be given to the supplier and the third set shall be kept under seal as a referee sample.

**7.2.1** All the moisture samples and the laboratory samples shall be preserved in dry, clean and well-stoppered bottles, labelled with full identification, such as the source and category of the mineral, the supplier's name, the lot number and the date of sampling. These samples shall be sent to a laboratory mutually agreed upon by the purchaser and the supplier for determination of chemical composition and the moisture content.

## 8. NUMBER OF TESTS

**8.1** All the laboratory samples for chemical analysis shall be tested individually for important characteristics; for the remaining, a composite sample shall be prepared by mixing equal quantities of the mineral from each of the laboratory samples and shall be analyzed. Those chemical characteristics for which each laboratory sample is to be analyzed and also those for which a composite sample is to be tested are given in Table 3 unless otherwise agreed between the purchaser and the supplier.

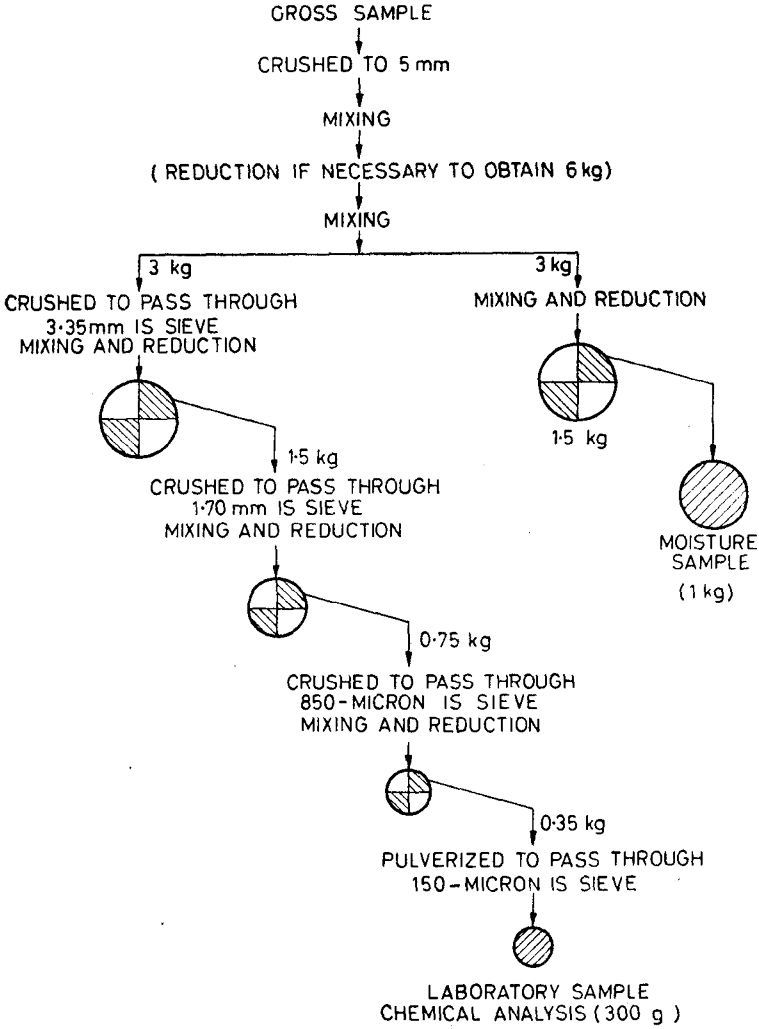
GROSS SAMPLE
CRUSHED TO 5 mm
MIXING
( REDUCTION IF NECESSARY TO OBTAIN 6 kg)
MIXING
3 kg
3 kg
CRUSHED TO PASS THROUGH 3·35mm IS SIEVE MIXING AND REDUCTION
MIXING AND REDUCTION
1·5 kg
MOISTURE SAMPLE (1 kg)
1·5 kg
CRUSHED TO PASS THROUGH 1·70 mm IS SIEVE MIXING AND REDUCTION
0·75 kg
CRUSHED TO PASS THROUGH 850-MICRON IS SIEVE MIXING AND REDUCTION
0·35 kg
PULVERIZED TO PASS THROUGH 150-MICRON IS SIEVE
LABORATORY SAMPLE CHEMICAL ANALYSIS (300 g )

FIG. 1 STAGES IN REDUCING A GROSS SAMPLE

## TABLE 3 NUMBER OF TESTS

( *Clause* 8.1 )

| Chemical Constituent of Cryolite for Which | | Chemical Constituent of Aluminium Trifluoride for Which | |
|---|---|---|---|
| Laboratory Samples are Analyzed Individualy | A Composite Sample is Analyzed | Laboratory Samples are Analyzed Individualy | A Composite sample is Analyzed |
| Total fluorine | $SiO_2$ | Total fluorine | $SiO_2$ |
| $CaF_2$ | $Fe_2O_3$ | | $Fe_2O_3$ |
| | Al | | $CaF_2$ |
| | Na | | $AlF_3$ |
| | Mg | | NaF |
| | Bound water | | Bound water |

## 9. REPORTING

### 9.1 Reporting of Chemical Composition

**9.1.1** For those characteristics, where a composite sample has been tested, only one test result will be available and that result shall be reported as the value of the characteristics for the lot sampled.

**9.1.2** When only one laboratory sample is available from a lot (that is, when the lot size is less than 100 tonnes), all the characteristics shall be determined on that sample and the results shall be reported as the value of the characteristics for the lot sampled.

**9.1.3** When only two laboratory samples have been analyzed individually from a lot, the average of the two available test results shall be reported as the value of the characteristic for the lot sampled. The individual test results shall also be reported to give an indication of the range of variation in quality.

**9.1.4** When three or more laboratory samples have been analyzed individually from a lot, the quality of the lot sampled with reference to each of the characteristics shall be reported as follows:

Let $x_1, x_2, x_3, \ldots\ldots\ldots x_n$ be the results of analyzing $n$ laboratory samples for a particular characteristic, then

Calculate

$$\text{Average } (\bar{x}) = \frac{(x_1 + x_2 + x_3 + \ldots\ldots\ldots\ldots x_n)}{n}$$

Range ( $R$ ) = the difference between the maximum and minimum values.

The average level of the characteristic in the lot shall be reported as equal to $\bar{x}$.

The limit of variation in the average level of that characteristic in the lot shall be reported as ( $\bar{x} \pm hR$ ) where $h$ is a factor, the value of which depends upon the number of laboratory samples analyzed. The appropriate value of the factor $h$ shall be taken from Table 4.

**TABLE 4 VALUES OF THE FACTOR *h***

| Number of Laboratory Samples Analyzed | Value of the Factor $h$ |
|---|---|
| 3 | 1·30 |
| 4 | 0·72 |
| 5 | 0·51 |
| 6 | 0·40 |

**9.2 Reporting of Moisture Content** — The results obtained by testing individually all the moisture samples representing a lot shall be reported in accordance with **9.1.2**, **9.1.3** or **9.1.4**.

( *Continued from page* 2 )

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Printed at Caslon Press, New Delhi 5, India